Rocky Mountain Conference on Magnetic Resonance

Volume 14 *14th Annual Rocky Mountain Spectroscopy Conference*

Article 1

July 1972

14th Annual Rocky Mountain Spectroscopy Conference

PROGRAM

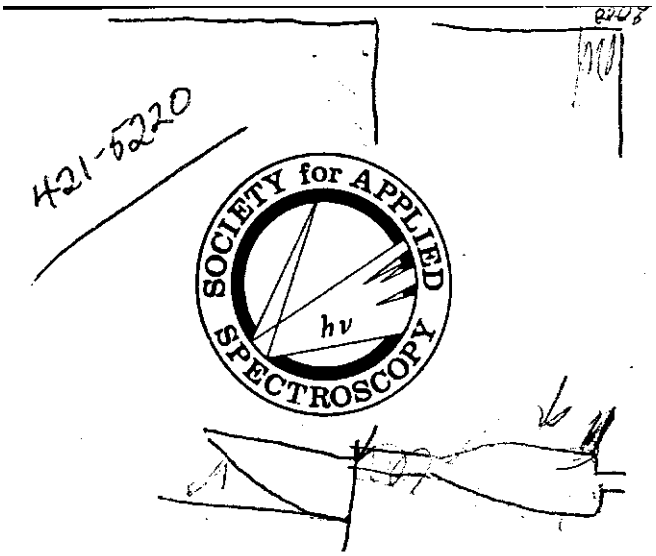
FOURTEENTH

ANNUAL

ROCKY MOUNTAIN

SPECTROSCOPY

CONFERENCE



July 31 - August 1, 1972

NEW ALBANY HOTEL
DENVER, COLORADO

Sponsored by the
Rocky Mountain Section
Society for
Applied Spectroscopy

Welcome to Denver

General Information

This year's conference continues the well established tone of the Rocky Mountain Spectroscopy Conferences in stressing quality papers, pertinent exhibits, a delightful banquet, and much friendly exchange in an easy, informal atmosphere. The papers of the program have been selected to cover a broad spectrum of the field called "Spectroscopy."

The Rocky Mountain Section of the Society for Applied Spectroscopy welcomes all of you to Denver and requests your full participation, so that everyone can have the desired happy — and worthwhile — experience.

Registration

Registration will begin at 7:45 a.m. on Monday, July 31, and continue throughout the conference. Checks should be made payable to the Rocky Mountain Section, Society for Applied Spectroscopy. The fees are as follows:

Members and Non-members	**$10.00**
Full time Students	**2.00**
Banquet	**6.00**

Registration badges will be required for admission to any session of the conference.

Accommodations

All technical sessions of the conference will be held in the Crown Jewel Room on the Mezzanine floor of the New Albany Hotel, 17th and Stout Streets.

Other nearby hotels are the Brown Palace, the Hilton, and the Cosmopolitan.

Other information on the Denver area may be obtained from the Denver Convention Bureau, 225 West Colfax Avenue, Denver, Colorado 80202.

Conference Mixer and Banquet

The hosted cocktail hour will be from 6 to 7 p.m. on Monday, July 31. The conference banquet will

be held at 7 p.m. that evening in the Crown Jewel Room. Banquet tickets may be purchased at the registration desk until Monday noon.

The Outstanding Service Award will be presented to a member of the RMS-SAS in recognition of service to the Rocky Mountain Section of the Society for Applied Spectroscopy.

Banquet Menu

Melon Ball Cocktail
Hearts of Lettuce Salad
Thousand Island Dressing
Roast U.S. Choice Prime Rib au Jus
Baked Potato
Buttered String Beans
Dinner Rolls
Fruit Tart
Coffee—Tea—Milk

Banquet Speaker

Mr. W. E. Davidson, Resident Manager of the NASA Skylab Program, will speak on "Skylab Overview." This will include an extremely interesting slide presentation with photos from manned space flights.

Coming originally from Dayton, Ohio, Bill is a 1951 graduate mechanical engineer of the University of Michigan at Ann Arbor. In the intervening 20 years, he has been closely associated with the missile and aerospace field, initially with Sandia Corporation's Rocket Sled Programs and Boeing's BOMARC Missile Program. For 17 years, he was with Dr. Wernher von Braun's NASA group at Huntsville, Alabama, where he was responsible for a variety of propulsion engineering systems on the Redstone and Jupiter missile and space programs. He was also responsible for several management and liaison activities associated with NASA's nuclear and Apollo manned rocket engines and spacecraft. Between '65 and '67, he served two years as City Councilman for Friendswood, Texas — a city near Houston, Texas, — and currently is on the Board of Trustees of Friends Bible College, Haviland, Kansas.

In September '67, Bill assumed the position as Skylab Program NASA Resident Manager to the Martin-Marietta Corporation representing both

the Marshall Space Flight Center, Huntsville, Alabama, and the Manned Spacecraft Center, Houston, Texas. Bill, his wife, and four children reside at Denver, Colorado.

Coffee Breaks

Coffee and rolls will be served at the morning breaks and coffee and soft drinks will be served at the afternoon breaks. These will be served in the exhibit area.

Contributors to the Coffee Break Fund are:

VWR Scientific
Blake Industries

Exhibits

Spectrographic equipment and materials will be exhibited in the Crown Jewel West room. The following companies will have booths in the exhibit area:

Beckman Instruments, Inc.
Canberra Industries
Continental Water Conditioning Corporation of Colorado
Kevex Corporation
Lab Test Equipment Company
Nicolet Instrument Corporation
Northern Scientific, Inc.
Packard Instrument Company
Technical Equipment Corporation

DRI X-Ray Conference

The 21st Annual Denver X-Ray conference on Applications of X-Ray Analysis will be held August 2-4, 1972, at the New Albany Hotel.

Monday Morning Session

Crown Jewel Room
July 31, 1972

Session Chairman — D. Blair Roberts, Westinghouse Georesearch Laboratory, Boulder, Colorado.

8:50 Welcome by RMS-SAS Chairman, David D. Conway

9:00 Mass Spectrometry of Metal Chelates. B. C. Wood, R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

9:20 Comparison of Sensitivity, Detection Limit and Interferences in Air-Acetylene and Nitrous Oxide-Acetylene Flames by Atomic Absorption Spectrometry. J. Y. Hwang, P. A. Ullucci, M. L. Smith, Instrumentation Laboratory Inc., Lexington, Massachusetts.

9:40 Systematics of Gaseous Atomic and Molecular Neutral and Ionic f-Values. R. Hefferlin, P. Brenneman, D. Durham, J. Flechas, Southern Missionary College, Collegedale, Tennessee.

10:00 The Application of Fission Tracks to Locate and Establish the Physical Form of Plutonium in Environmental Samples. J. A. Hayden, R. L. Murri, H. M. Baker, Dow Chemical U.S.A., Rocky Flats Division, Golden, Colorado.

10:20 Break.

10:40 Spectroscopic Identification of High-Boiling Pyridines in Shale-Oil Naphtha. F. R. McDonald, J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.

11:00 A Solid State Analysis of Mercuric Thiocyanates by X-Ray Flourescence. R. Botdorf, Wyomissing, Pennsylvania.

11:20 **A Computer Program for the Computation of Analytical Results from Atomic Absorption Data.** **O. K. Galle, L. M. Magnuson, Geochemistry Section, Kansas Geological Survey, University of Kansas, Lawrence, Kansas.**

11:40 **High Resolution ^{207}Pb Nuclear Magnetic Resonance.** **J. Dallas, G. E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado.**

12:00 **Lunch.**

Monday Afternoon Session

Crown Jewel Room
July 31, 1972

Session Chairman — Henry L. Dequasie, Delaware Valley Development and Service Corporation, Limon, Colorado.

1:30 **Improved Wavelength — Tunable Laser System for Infrared Spectroscopy in the *2\i* — 22 u-Spectral Region.** **C. F. Dewey, Jr., C. E. Hackett, Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.**

1:50 **A Spark Source Mass Spectrometric Examination of Environmental Impact of Oil Shale.** **W. R. Gilgren, C. G. Warren, M. L. Jacobs, Accu-Labs Research, Inc., Wheat Ridge, Colorado.**

2:10 **Experiences with the Carbon Rod Analyzer.** **D. L. Dick, R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado.**

2:30 **Break**

2:50 **Nuclear Magnetic Resonance Studies of Cholinergic Species.** **L. Simeral, G. E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado.**

3:10 **Prevention of Ejection of the Electrode Charge During Arcing of Uranium Compounds.** J. V. Pena, O. R. Simi, Los Alamos Scientific Laboratory, Los Alamos, New Mexico.

3:30 **A Comparator — Microdensitometer — Programable Calculator System for Processing High Resolution Mass Spectrographic Plates.** D. G. Earnshaw, J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.

3:50 **Problems Associated with the Utilization of Electrodeless Discharge Lamps as Sources for Atomic Absorption Spectroscopy.** K. E. Smith, W. G. Schrenk, Chemistry Department, Kansas State University, Manhattan, Kansas.

6:00 **Conference Mixer**

7:00 **Conference Banquet**
Speaker: Mr. W. E. Davidson, N.A.S.A.

Tuesday Morning Session

Crown Jewel Room
August 1, 1972

Session Chairman — A. P. Marranzino, U.S. Geological Survey, Denver, Colorado.

9:00 **Evaluation of the Chemical Analysis of the USGS Standards.** T. R. Copeland, J. L. Seeley, R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

9:20 **Factors Affecting the Resolution of Fine Structure in Electron Impact Ionization Efficiency Curves Obtained from Conventional Mass Spectrometers.** J. H. Weber, R. E. Ellefson, A. B. Denison, U.S. Department of the interior, Bureau of

Mines, Laramie Energy Research Center, Laramie, Wyoming.

9:40 The Analysis of Mercury in Biological Materials by Non-Flame Atomic Absorption Spectrophotometry. H. E. Taylor, M. L. Jacobs, R. Brown, Accu-Labs Research, Inc., Wheat Ridge, Colo.

10:00 Behind the Scenes in Spectroscopy — The Fourier Transformation and Some Manifestations. F. P. Miknis, J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.

10:20 Break

10:40 High-Resolution ^{199}Hg Nuclear Magnetic Resonance Spectroscopy. M. Borzo, G. E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado.

11:00 The Spectroscopic Investigation of Intramolecular Hydrogen-Bonding in Stereoisomeric Phenylhydrazones and Osazones. W. C. Stickler, W. C. Hoffman, Department of Chemistry, University of Denver, Denver, Colorado, L. L. Nolen, Chemistry Department, U.S. Air Force Academy, Colorado, L. N. Pflug, Seiler Research Laboratory, U.S. Air Force Academy, Colorado.

11:20 Computerized Computation of Analytical Results from Emission Spectrography. T. C. Waugh, L. M. Magnuson, Geochemistry Section, Kansas Geological Survey, University of Kansas, Lawrence, Kansas.

11:40 A Pulsed Nuclear Magnetic Resonance Spectroscopic Examination of Oil Shales. F. P. Miknis, A. W. Decora, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.

12:00 Lunch.

Tuesday Afternoon Session

Crown Jewel Room
August 1, 1972

Session Chairman — Stephen E. Valente, Chemistry Department, Regis College, Denver, Colorado.

1:40 **Vibrational Spectrum of N-chlorochloroformimidoyl Chloride. J. M. Burke, R. W. Mitchell, Physical Sciences Directorate, RDE&MSL, U.S. Army Missile Command, Redstone Arsenal, Alabama.**

Rec. Dir. Engr. + Miss Syst Lab.

2:00 **Quantitative Analytical Capability of an Energy Dispersive X-Ray Spectrometer with Multiple Single Channel Analyzers Using Electron Beam Excitation. G. A. Desborough, R. H. Heidel, U.S. Geological Survey, Denver Federal Center, Denver, Colorado.**

2:20 **Break.**

2:50 **Mass Spectral Pattern Recognition in Hydrocarbon Mixtures Using a Least Squares Approach, D. Fausett, D. G. Earnshaw, A. W. Decora, J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming.**

3:10 **Some Thoughts on Professional Unemployment Problems. H.L. Dequasie, Delaware Valley Development and Service Corporation, Millville, N.J., (Limon, Colorado).**

3:30 **Volatility Losses of Elements During Plasma Ashing: Mercury. R.M. Barnes, A. L. Bates, Department of Chemistry, University of Massachusetts, Amherst, Massachusetts.**

Rich

3:50 **Recent Developments in Silicon — 29 NMR. R. Elliott, G. E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado.**

Officers of the Rocky Mountain Section, Society for Applied Spectroscopy:

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Abstracts of Papers Presented

MONDAY

(9:00 a.m.) MASS SPECTROMETRY OF METAL CHELATES. Benny C. Wood and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Metal diethydithiocarbamates introduced into a mass spectrometer via the direct insertion probe can be quantitatively determined at low concentration levels. The method will be discussed and the general analytical utility considered.

(9:20) COMPARISON OF SENSITIVITY, DETECTION LIMIT AND INTERFERENCES IN AIR-ACETYLENE AND NITROUS OXIDE-ACETYLENE FLAMES BY ATOMIC ABSORPTION SPECTROMETRY. Jae Y. Hwang, Paul A. Ullucci, Marvin L. Smith, Instrumentation Laboratory Inc., 113 Hartwell Avenue, Lexington, Massachusetts 02173.

Those elements that can be determined with great sensitivity in air-acetylene flames by atomic absorption spectrometry have seldom been tested in nitrous oxide flames. However, in actual analyses, the demand for the use of nitrous oxide-acetylene flames has been increasingly stimulated because of the wide variety of sample matrices and unpredictable nature of the samples. In the present paper, a comparison is made on the sensitivity, detection limit, spectral and chemical interferences in both flames in an effort to expand the applicability of nitrous oxide-acetylene flames.

(9:40) SYSTEMATICS OF GASEOUS ATOMIC AND MOECULAR NEUTRAL AND IONIC f-VALUES. Ray Hefferlin, Pet Brenneman, Delynne Durham, and Jorge Flechas. Southern Missionary College, Collegedale, Tennessee 37315.

The f-value is related to the Einstein Anm the lifetime, the absorption coefficient, etc. The world supply of accurately measured and/or calculated atomic and ionic values is assembled and published by the NBS Atomic Data Center,

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and is still so limited that systematic study thereof usefully supplements new experiment and / or calculation. E.g., much has been learned from isoelectronic sequences.[1,2] In this presentation we describe results of comparisons of f-values along columns of the periodic table, e.g. the expected fact that for similar transitions between "high" "circular" orbits the f-values are comparable. We also discuss attempted comparisons of transitions in alkalies (jumping electron) and halogens (jumping hole?).[3] Due to a conspiracy of simple considerations there are practically no such pairs of f-values.

The world supply of diatomic molecular electronic f-values is very meagre, and existing collections consist of small lists of numbers or a couple of large bibliographies. Our comprehensive listing project is described, and also the planned critical evaluations. The objective is to make systematic comparisons analogous to those done for atoms and ions. It is an initial result that all f-values for a given electronic transition for all molecules with the same number of electrons are comparable in size.[4, 5]

1. Ray Hefferlin and Lucy Rascon, Paper BE 10, American Physical Society, Nashville, Tennessee, December 1, 1966; J. Opt. Soc. Am. 57,964 (1967)
2. M. W. Smith and W. L. Wiese, Astrophys. Journ. Supplement No. 196 (23, 103 (1971))
3. C. W. Ufford, Phys. Rev. 47 931 (1935)
4. Gerhard Herzberg, "Molecular Spectra and Molecular Structure I. Spectra of Diatomic Molecules," D. Van Nostrand, New York, 1950, page 386
5. H. E. Popkie and W. H Henneker, J Chem. Phys. 55, 617 (1971)

(10:00) THE APPLICATION OF FISSION TRACKS TO LOCATE AND ESTABLISH THE PHYSICAL FORM OF PLUTONIUM IN ENVIRONMENTAL SAMPLES.

J. A. Hayden, R. L. Murri, and H. M. Baker, Dow Chemical U.S.A., Rocky Flats Division, P.O. Box 888, Golden, Colorado 80401.

Exploratory experiments to find Pu and establish its physical form in environmental samples have been conducted. Types of samples examined include dust from laboratory and process area, outdoor air samples, soil and aquatic materials. Particle size data obtained from fission tracks are compared with electron microscope and autoradiographic results. Efforts to apply the technique to soluble forms of

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Pu in pond water and interferences from the presence of U are discussed.

(10:40) SPECTROSCOPIC IDENTIFICATION OF HIGH-BOILING PYRIDINES IN SHALE-OIL NAPHTHA. F. R. McDonald and J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

The identification of alkylpyridines in a heavy naphtha (150 to 280°C) fraction of thermally cracked shale oil requires careful correlation of spectral data. Infrared, nuclear magnetic resonance, and mass spectrometry were employed for the identification of individual pyridines in distillation fractions chromatographed on silica gel.

(11:00) A SOLID STATE ANALYSIS OF MERCURIC THIOCYANATES BY X-RAY FLUORESCENCE. Ruth Botdorf, 1701 Bern Road, Wyomissing, Pennsylvania 19610.

Fluorescence analysis is a nondestructive method which has been used in the quantitative determination of elements, both heavy and light, in substances such as minerals, alloys, and meteorites. It is a quite useful method when only very small quantities of sample are available. A single sample prepared in a disc can be analyzed for a variety of elements.

Cadmium, cobalt, copper, and zinc mercuric thiocyanates in addition to various combinations of any two of these compounds were prepared using potassium mercuric thiocyanate and metallic chloride solutions. Standards of cadmium, cobalt, copper, and zinc were made by fusing the metallic oxide with potassium pyrosulfate.

The intensity of reflected radiation emitted by the metal in each disc was measured with an x-ray fluorescence instrument incorporating a crystal of lithium fluoride. The percentage of metal in a mercuric thiocyanate was obtained from a calibration curve of percentage metal in a standard versus counts of radiation. The calibration curves were straight line relationships.

MONDAY

(11:20) A COMPUTER PROGRAM FOR THE COMPUTATION OF ANALYTICAL RESULTS FROM ATOMIC ABSORPTION DATA. O. Karmie Galle and Larry M. Magnuson, Geochemistry Section, Kansas Geological Survey, University of Kansas, Lawrence, Kansas 66044.

A computer program to aid in the calculation of analytical results from atomic absorption data is described. The program averages a desired number of absorption readings for each standard and unknown solution, converts the absorption to absorbance, plots a least squares fit of the standard curve, calculates the results, and prints them out in tabular form. The program is also designed to print out, if desired, a plot of the standard curve as well as a statistical analysis of the curve such as goodness of fit and standard deviation. Written in BASIC language, the program can be used with a remote terminal hook-up to a large central computer facility. Also described, is a separate program designed to monitor the A.A. data read into the first program to assure that the correct number of readings are entered and properly sequenced. This second program was written to locate format errors and prevent premature termination of program execution due to improperly entered data. Advantages of the programs will be discussed and examples of how the data is entered as well as print-outs of tabulated results, standard curves, and statistical data will be shown.

(11:40) HIGH RESOLUTION ^{207}Pb NUCLEAR MAGNETIC RESONANCE. Jerry Dallas and Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Using pulse fourier transform techniques, the nuclear magnetic resonance of ^{207}Pb has been investigated in a representative series of organolead compounds. Some aspects of the instrumental approach that is employed in this work will be briefly reviewed. Experimental results will be discussed with particular regard to the structural dependence of ^{207}Pb chemical shifts.

A brief discussion will also be included regarding the feasibility and prospects of

quantitative applications of this technique to current and future analytical problems.

(1:30 p.m.) IMPROVED WAVELENGTH-TUNABLE LASER SYSTEM FOR INFRARED SPECTROSCOPY IN THE 2u. - 22u. Spectral Region*. C. Forbes Dewey, Jr., and Colin E. Hackett, Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass. 02139.

A laser system for producing wavelength-tunable infrared radiation was described in a previous paper.[1] In this paper we reported significant increases in the spectral coverage of this system using the nonlinear optical materials proustite (Ag_3AsS_3), cinnabar (HgS), and the trigonal form of Selenium. Theoretical calculations of phase-matching conditions and expected infrared power will be presented.

A pulsed ruby laser-dye system can produce up to 100 KW of continuously-tunable infrared radiation in the 2u. - 5u. region, and between 10 KW and 100W of pulsed power between 5u, and 22u..

Considerations influencing the spectral width of the infrared radiation will be discussed. The use of an intra-cavity Brewster-angle prism appears most attractive for continuous wavelength tuning with a resolution of about 3 cm^{-1}. By introducing a 3-element Lyot filter, the resolution can be improved to 0.2cm^{-1}, while maintaining continuous tuning capability.

Additional advances to be reported are: polymethine laser dye solutions with improved temporal stability; and a dye laser optical configuration with increased pumping efficiency and uniform excitation.

*This research was supported by the Advanced Research Projects Agency under Contract DAHCO4-71-C-0049.

1. C. F. Dewey, Jr., paper presented at the 13th Annual Rocky Mountain Spectroscopy Conference, August 9-10; 1971.

(1:50) A SPARK SOURCE MASS SPECTROMETRIC EXAMINATION OF ENVIRONMENTAL IMPACT OF OIL SHALE. W. R. Gilgren, C. G. Warren, M. L. Jacobs, Accu-Labs Research, Inc., 9170 W. 44th Avenue, Wheat Ridge, Colorado 80033.

A spark source mass spectrometric survey

analysis of raw and spent oil shale has been carried out to determine if elements present may constitute a health hazard.

A similar examination of the composition of various aqueous extracts from spent oil shale has also been carried out to determine if water elution of toxic elements from spoil heaps will constitute a problem.

A brief description of the technique and applicability of the method to this type of analysis will be given.

(2:10) EXPERIENCES WITH THE CARBON ROD ANALYZER. D. L. Dick and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

The direct determination of trace elements by atomic absorption is often precluded by a lack of sensitivity and/or the unavailability of adequate sample. The carbon rod analyzer offers some potential for alleviation of such problems. A number of specific experiences with this system will be discussed and an evaluation of its capabilities and limitations presented.

(2:50) NUCLEAR MAGNETIC RESONANCE STUDIES OF CHOLINERGIC SPECIES. Larry Simeral and Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Proton and carbon-13 magnetic resonance have been used to explore the conformational and electronic properties of molecules which are pharmacologically active on the cholinergic nervous system. Species known to be active toward muscarinic and nicotinic receptors and acetylcholinesterase have been investigated. The results indicate similar conformations and electron distributions for nicotine and acetylcholine in aqueous solution. The results for compounds such as atropine, oxotremorine and their analogs will be discussed in terms of structure-activity relationships.

(3:10) PREVENTION OF EJECTION OF ELECTRODE CHARGE DURING ARCING OF

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URANIUM COMPOUNDS*. Juanita V. Pena and Oliver R. Simi, Los Alamos Scientific Laboratory, Los Alamos, N. M. 87544.

The ejection of the charge from the electrode crater during arcing has long been a problem to spectrochemists. The problem is particularly serious when alloys of uranium are being analyzed by the carrier distillation technique.' These ejections can be essentially prevented by drilling small (0.46 mm diameter) holes in the electrode at the level of the charge, and by ensuring very good electrical contact between the electrode and the circuit. Other preventative techniques as well as the history of these ejections will be discussed.

*Work performed under the auspicies of the U.S. Atomic Energy Commission.

1. Scribner, B. F. and Mullins, H. R., "Carrier Distillation Method for Spectrographic Analysis and its Application to Uranium-Base Materials," J. Research NBS, 37, 379 (1946).

(3:30) A COMPARATOR-MICRODENSITOMETER-PROGRAMABLE CALCULATOR SYSTEM FOR PROCESSING HIGH RESOLUTION MASS SPECTROGRAPHIC PLATES. D. G. Earnshaw and J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

The hardware system and software package for the semi-automatic acquiring and processing of high resolution mass spectrographic plates is described. The semi-automatic data acquisition part of the system allows a time savings of 200 percent as compared to the hand read data acquisition. Processing of the high resolution data to yield exact masses and the corresponding empirical formulas is accomplished by efficient programming on the small, programable calculator.

(3:50) PROBLEMS ASSOCIATED WITH THE UTILIZATION OF ELECTRODELESS DISCHARGE LAMPS AS SOURCES FOR ATOMIC ABSORPTION SPECTROSCOPY. K. E. Smith and W. G. Schrenk, Chemistry Department, Kansas State University, Manhattan, Kansas 66506.

The use of electrodeless discharge lamps

MONDAY

(EDL's) as spectral source units for AAS is complicated by many constructional parameters. Many of these can presently be determined only through trial-and-error evaluation. Since these parameters are interrelated this process is usually quite tedious.

This paper will be a discussion of the problems involved in optimizing many of the EDL's parameters. These include the size of the lamp, coupling of the microwave radiation to the lamp, regulation of the lamp's operating temperature, spectral broadening of the emission lines, the nature of the fill gas and the fill gas pressure.

TUESDAY

(9:00) EVALUATION OF THE CHEMICAL ANALYSIS OF THE USGS STANDARDS. T. R. Copeland, J. L. Seeley, and R. K. Skogerboe, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Although the collaborative analysis results of the U.S.G.S. standards were published several years ago, an evaluation of those results has not appeared. Examination of the results readily indicates that a number of the reporting laboratories produced results subject to serious systematic errors and that these erroneous results have been used in computing the recommended concentration values. An objective statistical approach has been used to isolate those results subject to systematic errors and eliminate them from consideration in determining the "true" concentrations. The method used and a general evaluation of the results will be presented.

(9:20) FACTORS AFFECTING THE RESOLUTION OF FINE STRUCTURE IN ELECTRON IMPACT IONIZATION EFFICIENCY CURVES OBTAINED FROM CONVENTIONAL MASS SPECTROMETERS. J. H. Weber, R. E. Ellefson and A. B. Denison, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

The energy distribution difference (EDD) technique for resolving fine structure in

ionization efficiency curves is reviewed in terms of its application to conventional mass spectrometers. We have found that regulation of the ionizing electron beam to give a constant current either by varying the filament temperature or by use of a control grid bias changes the shape of the thermal energy distribution of the ionizing electrons. The change of this thermal distribution introduces errors in appearance potentials when using control grid bias regulation and errors in relative transition probabilities when using filament temperature regulation. Convolution and deconvolution studies of ionization efficiency curves suggest that the ionizing electron beam current should be unregulated to maintain a constant energy distribution and that corrections be made for the resulting change in the ionizing electron beam current with accelerating voltage.

(9:40) THE ANALYSIS OF MERCURY IN BIOLOGICAL MATERIALS BY NON-FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY. H. E. Taylor, N. L. Jacobs, R. Brown.

Techniques are described for the direct analysis of mercury in biological materials without tedious sample preparation. Both solid and liquid samples are analysed utilizing a direct combustion method.

Typical analyses of human biological material such as lung, liver, serums, and whole blood are discussed. Analyses of other biological materials such as fish, milk, plants, and soils are also presented.

(10:00) BEHIND THE SCENES IN SPECTROSCOPY — THE FOURIER TRANSFORMATION AND SOME MANIFESTATIONS. F. P. Miknis and J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

The use of the technique of Fourier transformation (FT) is undergoing a spectroscopic renaissance, owing to its ever increasing applications in the fields of nuclear magnetic resonance (NMR) and infrared (IR) spectroscopy. This paper reviews the basic principles of FT theory and its spectroscopic

manifestations. It is shown that the FT as it is used in NMR and IR applications follows the general philosophy of transformation theory in problem solving.

(10:40) HIGH-RESOLUTION Hg NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY. Marie Borzo and Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Pulse Fourier transform methods have been applied to the case of ^{199}Hg in organomercury and inorganic species. A wide range of chemical shifts and linewidths have been observed. Instrumental approaches and prospects for applications of ^{199}Hg in structural and analytical problems will be discussed.

(11:00) THE SPECTROSCOPIC INVESTIGATION OF INTRAMOLECULAR HYDROGEN-BONDING IN STEREOISOMERS PHENYLHYDRAZONES AND OSAZONES. W. C. Stickler, W. C, Hoffman, Department of Chemistry, University of Denver, Denver, Colorado 80210, L. L. Nolen, Chemistry Department, U.S. Air Force Academy, Colorado 80840, L. N. Pflug, Seiler Research Laboratory, U.S. Air Force Academy, Colorado 80840.

Structural prerequisites for the occurrence and isolation of geometrical isomers of phenylhydrazones are briefly discussed. Intramolecular hydrogen-bonding is a major factor in their occurrence and has been investigated with the aid of IR and NMR spectroscopy. Position and intensity of bands will be illustrated with a number of examples (such as the phenylhydrazones of benzoin and 2-benzoylpyridines and the p-nitrophenylhydrazones of benzoin). The N-H • • • N intramolecular hydrogen bridge plays an important role in bisphenylhydrazones (osazones); of 16 osazones studied to date, 12 possess the amphi configuration (with cis conformation), exhibiting N-H • • N bonding.

(11:20) COMPUTERIZED COMPUTATION OF ANALYTICAL RESULTS FROM EMISSION

TUESDAY

SPECTROGRAPHY. Truman C. Waugh and Larry M. Magnuson, Geochemistry Section, Kansas Geological Survey, University of Kansas, Lawrence, Kansas 66044.

Three time-sharing computer programs for reduction and evaluation of emission spectrographic data are described. These programs are written in BASIC language for use with remote terminal facilities. The first program performs a least squares fit on data derived from the film and establishes pre-calibration and calibration curves closely following ASTM Method E 116-70a and stores coefficients to be used by the following two programs. The second program pre-processes transmission values from standards, calculates analytical curves by least squares analysis, and stores coefficients for use by the last program. The third program converts transmission data from samples to relative intensity values and determines concentrations from analytical curves. Analytical results and statistical evaluations are tabulated in a form suitable for final report. This system is designed for maximum flexibility, reduces time required in handling emission spectrographic data, allows large amounts of data to be processed, and improves accuracy and precision.

(11:40) A PULSED NUCLEAR MAGNETIC RESONANCE SPECTROSCOPIC EXAMINATION OF OIL SHALES. F. P. Miknis and A. W. Decora, U. S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070.

Previous studies in our laboratory showed that wide-line nuclear magnetic resonance (NMR) spectroscopy has potential applications in such areas as predicting oil yields and organic carbon contents of oil shales. In a continuation of this study the techniques of pulsed NMR have been applied to the measurements of hydrogen content and spin-lattice,^, relaxation times in oil shales. The measurements of hydrogen content linearly correlated with Fischer assay oil yields, thus enabling the use of pulsed NMR as a rapid, non-destructive assay method for oil shales. These results were based on pulsed measurements for 140 samples taken from a recently Fischer assayed test-lease core. The

spin-lattice relaxation times were measured on a different set of shale samples. A *90°T* 90° pulse sequence was used for these measurements and the T]'s ranged from about 1 msec, to 200 msec, with most shales exhibiting multiple relaxations. These data may prove useful in the determination of different types of protons in oil shales.

(1:40) VIBRATIONAL SPECTRUM OF N-CHLOROCHLOROFORMIMIDOYL CHLORIDE. J. Michael Burke and Ronald W. Mitchell, Physical Sciences Directorate, RDE&MSL, U.S. Army Missile Command, Redstone Arsenal, Alabama 35809.

N-chlorochloroformimidoyl chloride (Cl_2CNCl) was prepared by the method of Hagemann, et al. and purified by distillation. Infrared spectra (250 - 4000 cm') have been obtained on vapor, liquid, and solid phase samples of this molecule. The Raman spectrum of a liquid sample has also been recorded and polarization data obtained. Vapor phase band contours, Raman polarization data, and characteristic group frequencies have been utilized in making the vibrational assignments which will be discussed.

(2:00) QUANTITATIVE ANALYTICAL CAPABILITY OF AN ENERGY DISPERSIVE X-RAY SPECTROMETER WITH MULTIPLE SINGLE CHANNEL ANALYZERS USING ELECTRON BEAM EXCITATION. George A. Desborough and Robert H. Heidel, U.S. Geological Survey, Denver Federal Center, Denver, Colo. 80225.

Rapid, non-destructive, multiple and simultaneous quantitative elemental analysis of K, L, and M X-ray spectra in the energy range of about 1.74 keV (Si Ka,p) to 8.64 keV (Zn Ka) of elements in major concentrations for solid materials is accomplished utilizing a stable electron beam of intensity less than 10 nanoamperes specimen current at operating voltages of 10 to 20 kV. Appropriate reference standards are used to illustrate quantitative chemical determinations for simple binary and ternary systems using Ka.p, La, and/or Ma spectra. Other rapid analysis (10 sec.)

semiquantitative and qualitative applications are illustrated. The major impediment to quantitative analysis is the correction or uncertainty due to background effects.

(2:50) MASS SPECTRAL PATTERN RECOGNITION IN HYDROCARBON MIXTURES USING A LEAST SQUARES APPROACH. D. Fausett, D. G. Earnshaw, A. W. Decora and J. H. Weber, U.S. Department of the Interior, Bureau of Mines, Laramie Energy Research Center, Laramie, Wyoming 82070

A method for determining, by low-resolution mass spectroscopy, the constituent compounds and their relative concentrations in a mixture of hydrocarbons is presented. The analytical method employed assumes that a reference set of mass spectra of pure compounds is available which contains the spectra of the dominant compounds present in the mixture. The analytical method involves identifying a subset of the reference compounds whose spectra generate the best least squares fit to the spectrum of the mixture in comparison with other possible subsets. Several approaches to this problem as well as examples of results obtained are presented.

(3:10) SOME THOUGHTS ON PROFESSIONAL UNEMPLOYMENT PROBLEMS. Henry L. Dequasie, Delaware Valley Development & Service Corp., Millville, N. J. (P.O. Box 356, Limon, Colorado 80828).

Relief of unemployment in professional categories is a complex problem because of the many specialized criteria that must be satisfied in establishing an employer-employee relationship. The job-search methods usually available to an unemployed person are not very effective or satisfactory in engineering and science fields because of limited area-coverage by individual efforts and the incompetent, indifferent, or too costly assistance by employment agencies. However, the methods of private employment agencies, applied by groups within professional societies, could be used to locate suitable positions for unemployed members and, in addition, be a financially self-supporting operation.

TUESDAY

(3:30) VOLATILITY LOSSES OF ELEMENTS DURING PLASMA ASHING: MERCURY. Ramon M. Barnes and Anne L. Bates, Department of Chemistry, University of Massachusetts, Amherst, Massachusetts 01002.

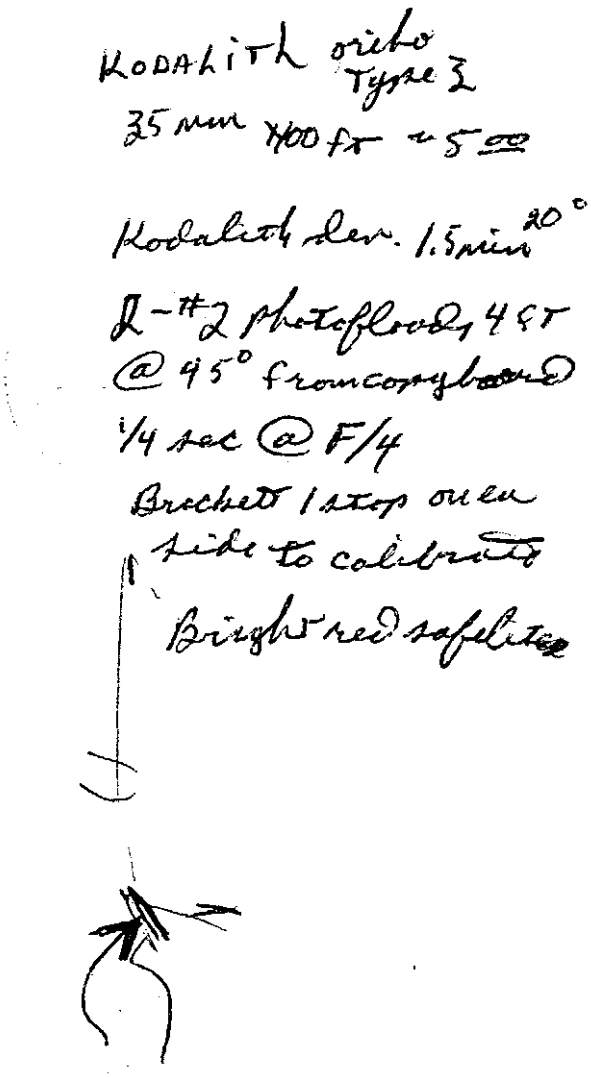
The low-temperature plasma ashing method for sample preparation retains inorganic but removes organic material by reaction of activated oxygen in a flowing, low-pressure, radio frequency discharge. Because of the relatively mild, contamination-free conditions, plasma ashing is ideal for specimens containing volatile metals often lost completely in harsh ashing methods.

This report presents preliminary results for plasma ashing mercury-containing samples. Mercury volatilization was followed by emission and absorption spectrometry during ashing, and rates were determined at different operating conditions of pressure, pumping rate, generator power, oxygen flow, and organic matrix for inorganic and organic mercury compounds. For widely varying conditions, mercury loss appears complete during ashing. New approaches to reduction of mercury volatilization rates will be presented.

Using conventional spectrometry, quantitative mercury analysis appears feasible during the ashing of a sample that will be used for subsequent metal analysis.

(3:50) RECENT DEVELOPMENTS IN SILICON-29 NMR. Richard Elliott and Gary E. Maciel, Department of Chemistry, Colorado State University, Fort Collins, Colorado 80521.

Pulse Fourier transform as well as continuous wave techniques have been used for silicon NMR. Structure vs_shift relationships as well as the relationship between structure and silicon nuclear Overhauser effects have been examined in some organosilicon compounds. Contributions to the 29SJ spin-lattice relaxation times for some compounds have been investigated. The general features of the experimental techniques will be discussed.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